

s10.m2.p3 **Structural Studies of Ultrafine-Grained Materials Obtained by Severe Plastic Deformation.** R. Kužel¹, R.K. Islamgaliev², J. Cížek¹, F. Chmelik¹, ¹*Faculty of Mathematics and Physics, Charles University, Prague, Czech Republic,* ²*Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa, Russia.*

Keywords: powder diffraction, microstructure analysis.

Severe plastic deformation is a technique capable of producing ultrafine-grained (UFG) samples of high purity and no residual porosity [1] which are suitable for studies by different techniques.

Two kind of materials were investigated – semiconductors (silicon, germanium) and f.c.c. metals (copper, nickel, copper with 0.5% Al₂O₃). X-ray diffraction (XRD), transmission electron microscopy, differential scanning calorimetry (DSC), life-time positron spectroscopy, microhardness measurements and other techniques were applied. The temperature evolution of microstructure and phase composition of the materials were studied and temperatures of changes were determined. This means first of all the relaxation of the grain-boundary (GB) dislocations and grain growth.

Positron spectroscopy indicated the presence of dislocations and microcavities of the size of about 5 monovacancies. It was confirmed that Al₂O₃ stabilizes the UFG structure of copper

XRD patterns of Si and Ge were characteristic by the presence of high-pressure phases which disappeared at higher temperatures, large XRD line broadening indicating the presence of microstrain of about 1 % and coherent domain size less than 10 nm, weak texture and no line-broadening anisotropy. The patterns of all copper and nickel UFG samples show no other phases, the broadening is smaller (strains < 0.1 %, domain size > 50 nm) but significant line-broadening anisotropy of the type h00 > hhh is always present. This was also found on Cu and Ni milled powders. The anisotropy is not significantly dependent on the orientation of the measured planes with respect to the surface as it was confirmed by the measurement at different inclinations. Cu and Ni samples have no or weak textures (of both (111) and (100) types). Lattice parameters coincide with the value given in the ICDD PDF-2 database.

The anisotropy of line widths can be in principle explained by the dislocation-induced broadening and orientation factors derived by Ungar et.al. [2]. However, the agreement is not complete and more theoretical considerations should be done. The problems probably arise from the strongly inhomogeneous distribution of dislocations which form large-angle grain boundaries with random misorientations.

Anyway, XRD can give much valuable information on thermal evolution of the microstructure of UFG materials and is well complementary to other techniques which are sensitive to different microstructural features.

[1] R.Z. Valiev, *Nanostructured Materials*, (1995) 6: 73 -

[2] T. Ungar, I. Dragomir, A. Revesz, A. Borbely, *J. Appl. Cryst.* (1999) 32:992-1002.

s10.m2.p4 **XRD study of size effects in Ni nanoparticles.** L.M. Plyasova, T.A. Kriger, I.Yu. Molina, M.A. Ermakova and V.N. Parmon, *Boriskov Institute of Catalysis, Pr. Akademika Lavrentieva, 5 Novosibirsk, Russia, 630090. E-mail: pls@catalysis.nsk.su*

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It is known that an abnormal phase state, specific morphological structures, defective atomic structures can be achieved for small particles. Therefore, physicochemical properties of nanoparticles differ from those of bulky materials. Besides, the size effect is the factor influencing catalytic properties of nanoparticles. We conducted XRD studies of Ni catalysts for methane decomposition. The catalyst lifetime was found to depend on the average size of catalytic nanoparticles.

Nickel catalysts were prepared by the sol-gel heterophase synthesis [1]. This method allow the average size catalytic nanoparticles to be varied in the range of 5 to more than 100 nm at the same loading of the active component in catalysts, the minimal interaction of the active component with the carrier being observed.

X-ray diffraction patterns were recorded in the scanning mode using a diffractometer URD-63 (Germany) with monochromatic Cu K_α-radiation. The studies were aimed at analysis of line broadening, determination of the lattice constants, the real structure and thermal fluctuations amplitude. The software used for simulation of X-ray diffraction profiles of polycrystalline materials including imperfections of different kinds is described elsewhere [2].

The size effects was shown to reveal in the samples under study when the particles were less than 10 nm in size. For these particles, the stacking faults along the [111] direction of the fcc structure of nickel, increased mean lattice constants and, correspondingly, a longer average Ni-Ni distance were observed. As a result, the amplitude of thermal fluctuations increased and the Debye temperature decreased. The obtained structural data must be taken into account for thermodynamic calculations and analysis of physicochemical properties of catalysts.

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[1] M.A. Ermakova, D.Yu. Ermakov, G.G. Kuvshinov, L.M. Plyasova, *J. Catal.*, 187 (1999) 77-84.

[2] S.V. Cherepanova, S.V. Tsybulya, *ProcEPDIC-5*, Parma, Italy, 1997, p.60